Patent Claims

1. Process for producing a compound of the formula $LiMPO_4$, in which M represents at least one metal from the first transition series, comprising the following steps:

a. production of a precursor mixture, containing at least one Li^+ source, at least one M^{2+} source and at least one PO_4^{3-} source, in order to form a precipitate and thereby to produce a precursor suspension;

b. dispersing or milling treatment of the precursor mixture and/or the precursor suspension until the D90 value of the particles in the precursor suspension is less than 50 μm;

c. the obtaining of $LiMPO_4$ from the precursor suspension obtained in accordance with b), preferably by reaction under hydrothermal conditions.

2. Process according to Claim 1, characterized in that the D90 value of the particles in the suspension is at most 25 μm, in particular at most 20 μm, particularly preferably at most 15 μm.

3. Process according to Claim 1 or 2, characterized in that M at least comprises Fe or represents Fe.

4. Process according to one of the preceding claims, characterized in that M comprises Fe, Mn, Co and/or Ni.

5. Process according to one of the preceding claims, characterized in that the $LiMPO_4$ is obtained in pure-phase form.

6. Process according to one of the preceding claims,

characterized in that the dispersing or milling treatment is used before or during the precipitation of the precursor mixture and is continued until the precipitation has concluded.

7. Process according to one of the preceding claims, characterized in that the dispersing treatment is used before the precipitation of the precursor mixture, in order to ensure a high level of crystal nucleation and to prevent the formation of large crystals and crystal agglomerates.

8. Process according to one of the preceding claims, characterized in that no evaporation occurs prior to the reaction of the precursor mixture or suspension under hydrothermal conditions.

9. Process according to one of the preceding claims, characterized in that no sintering takes place prior to the reaction of the precursor mixture or suspension under hydrothermal conditions.

10. Process according to one of the preceding claims, characterized in that the $LiMPO_4$ is dried following the reaction under hydrothermal conditions.

11. Process according to one of the preceding claims, characterized in that the production of the precursor mixture or suspension or the conversion under hydrothermal conditions takes place in the presence of at least one further component, in particular a carbon-containing or electron-conducting substance or the precursor of an electron-conducting substance.

12. Process according to one of the preceding claims, characterized in that the electron-conducting substance is carbon, in particular conductive carbon or carbon fibres.

13. Process according to one of the preceding claims, characterized in that the precursor of an electron-conducting substance is a carbon-containing substance, in particular a sugar compound.

14. Process according to one of the preceding claims, characterized in that the lithium source used is LiOH or Li_2CO_3.

15. Process according to one of the preceding claims, characterized in that the Fe^{2+} source used is a Fe^{2+} salt, in particular $FeSO_4$, $FeCl_2$, $FeNO_3$, $Fe_3(PO_4)_2$ or an organyl salt of iron.

16. Process according to one of the preceding claims, characterized in that the PO_4^{3-} source used is phosphoric acid, a metal phosphate, hydrogen phosphate or dihydrogen phosphate.

17. Process according to one of the preceding claims, characterized in that water is used as solvent in the precursor mixture.

18. Process according to one of the preceding claims, characterized in that the Li^+ source, the M^{2+} source are used in the form of aqueous solutions, and the PO_4^{3-} source is used in the form of a liquid or an aqueous solution.

19. Process according to one of the preceding claims, characterized in that the precipitate formed in the precursor suspension comprises at least one precursor of $LiMPO_4$, in particular vivianite, and the reaction to form $LiMPO_4$ then preferably takes place under hydrothermal conditions.

20. Process according to one of the preceding claims, characterized in that temperatures of between 100 and 250°C, in particular from 100 to 180°C, and a pressure

of from 1 bar to 40 bar, in particular from 1 bar to 10 bar steam pressure, are used under the hydrothermal conditions.

21. Process according to one of the preceding claims, characterized in that the components of the precursor mixture are present in the following stoichiometric ratio:

a. 1 mole Fe^{2+} : 1 mole PO_4^{3-} : 1 mole Li* (1:1:1)
b. 1 mole Fe^{2+} : 1 mole PO_4^{3-} : 3 mol Li* (1:1:3)
c. any mixing ratio between a and b.

22. Process according to one of the preceding claims, characterized in that the combining or reaction of the precursor mixture or suspension under hydrothermal conditions takes place under an inert gas atmosphere, preferably in the same vessel.

23. Process according to one of the preceding claims, characterized in that first of all, in an aqueous solvent, the M^{2+} source and the PO_4^{3-} source are mixed, in particular under an inert gas atmosphere, then, preferably once again under a protective gas or inert atmosphere, the Li^+ source is added, and then the reaction under hydrothermal conditions is carried out.

24. Process according to one of the preceding claims, characterized in that the dispersing or milling treatment is a treatment with dispersing means (with or without pump rotor), Ultraturrax, mills such as colloid mills or Manton-Gaulin mills, intensive mixers, centrifugal pumps, in-line mixtures, mixing nozzles, such as injector nozzles, or ultrasound appliances.

25. Process according to one of the preceding claims, characterized in that a stirring mechanism or the like, in particular an Ultraturrax stirrer, is used for the high-shearing treatment in accordance with Claim 1b,

with the introduction of power, calculated according to the formula $P = 2 \pi n M$, where M represents the torque and n represents the rotational speed, being at least 5 kW/m^3, in particular at least 7 kW/m^3.

26. Process according to one of the preceding claims, characterized in that the additional component in accordance with Claim 11 or 12 is used as a crystallization nucleus in the precipitation or reaction of the precursor mixture.

27. $LiMPO_4$, in particular $LiFePO_4$, obtainable by one of the preceding process claims.

28. $LiMPO_4$, in particular according to Claim 27, characterized in that the mean particle size (D50 value) is less than 0.8 μm, preferably less than 0.7 μm, in particular less than 0.6 μm, particularly preferably less than 0.5 μm.

29. $LiMPO_4$, according to Claim 27 or 28, characterized in that the D10 value of the particles is less than 0.4 μm, preferably less than 0.35 μm, and also preferably the D90 value is less than 3.0 μm, in particular less than 2.5 μm, most preferably less than 2.0 μm.

30. $LiMPO_4$, according to one of Claims 27 to 29, characterized in that the difference between the D90 value and the D10 value of the particles is no more than 2 μm, preferably no more than 1.5 μm, in particular no more than 1 μm, particularly preferably no more than 0.5 μm.

31. $LiMPO_4$, according to one of Claims 27 to 30, characterized in that the BET surface area is more than 3.5 m^2/g, in particular more than 4 m^2/g, particularly preferably more than 5 m^2/g, further preferably more than 10 m^2/g, most preferably more than 15 m^2/g.

32. Composition, in particular electrode material, containing $LiMPO_4$ according to one of Claims 27 to 31.

33. Composition according to Claim 32, also containing at least one further component, in particular a carbon-containing or electron-conducting substance or the precursor of an electron-conducting substance, particularly preferably carbon, conductive carbon or carbon fibres.

34. Secondary battery, containing a composition, in particular an electrode material according to Claim 32 or 33.

35. Use of $LiMPO_4$ according to one of Claims 27 to 31 as electrode material.

36. Process according to one of Claims 1 to 26, characterized in that the $LiMPO_4$, after the hydrothermal treatment, is separated off, in particular by filtration and/or centrifuging, if appropriate dried and if appropriate deagglomerated.

37. Process according to one of Claims 1 to 26, characterized in that the $LiMPO_4$ obtained from the hydrothermal treatment in a pyrolysis process, in which at least one carbon precursor material, preferably a carbohydrate, such as sugar or cellulose, and particularly preferably lactose, is mixed with the $LiMPO_4$, e.g. by kneading, it being possible to add water as an auxiliary.

38. Process according to Claim 37, characterized in that the carbon precursor material is added to the moist $LiMPO_4$ filter cake obtained by separation after the hydrothermal synthesis, the mixture of $LiMPO_4$ and carbon precursor material is dried and heated to a temperature between 500°C and 1000°C, preferably

between 700°C and 800°C, during which operation the carbon precursor material is pyrolyzed to form carbon.

39. Process according to Claim 38, characterized in that the pyrolysis is followed by a milling or deagglomeration treatment.

40. Process according to Claim 38 or 39, characterized in that the drying is preferably carried out under protective gas, in air or in vacuo at temperatures of preferably from 50°C to 200°C, and the pyrolysis is carried out under protective gas.